Australasian Soil and Plant ANALYSIS Council Inc.

ASPAC Digest – May 2018

11th Edition, May 2018

Contents

Australasian Soil and Plant Analysis Council Inc.
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ASPAC
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Greetings from the Chair

Dave Lyons – Chairman ASPAC, Queensland Representative

Welcome to readers of this ASPAC Newsletter for May 2018. We are running a little late with this edition as we wanted to first sure up the arrangements for ASPAC's participation in the National Soil Conference in Canberra 2018 to give you complete information. We are a kindred organisation with Soil Science Australia who are hosting this conference and we have been working with them to give ASPAC members the opportunity to contribute to the conference with the inclusion of topics relevant to our organisation. We have two members assisting the organising committee, Janice Trafford and myself.

Also in this edition we have information on the New Zealand Society of Soil Science Conference in Napier 2018. We have produced a report titled "ASPAC certification of laboratory proficiency in soil and plant testing - how it is assessed and what it means" that was sent to Fertiliser Australia, who were after information on this topic. This contains useful information that members can use in discussions with customers and collaborators and possibly management. Members of the ASPAC Executive and the Laboratory Proficiency Committee (LPC) contributed to this report. We can announce the winner of the Janice Trafford Travel Award for 2018 as well as an an artide prepared by the ASPAC methods sub-committee titled "Exchangeable cations method selection".

Conferences

KEY DATES

Abstract Submission
06 July 2018 - *Abstract Submissions close*
http://nzssconference.co.nz/

Registration online
20 October 2018 - *Early bird registrations close*

This conference will be held from 3rd to 6th of December. Rebecca Withnall our New Zealand representative on the ASPAC Executive, is on the organising committee and can take any enquiries from members intending to go to this conference (rebecca.withnall@datanow.co.nz.) ASPAC is sponsoring an activity at the conference.

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SOIL SCIENCE AUSTRALIA

NATIONAL SOILS CONFERENCE
Canberra 18-23 November 2018

SOIL: The key to the Past, the Present and the Future

KEY DATES

Abstract Submission
18 June 2018 - *Abstract Submissions close*

Registration
27 August 2018 - *Early bird registrations close*
12 November 2018 - *Online registrations close*
Note: *ASPAC members should select the member options when registering as they are to pay the same as Soil Science Australia members, and as such a significant discount will apply*

http://soilscienceconference.org.au/

ASPAC members will be leading two session topics, which will run on Thursday 22nd. *If you are attending, please consider presenting a paper in one of these topics or in any of the other twenty three. – see website*

Phil Moody will lead session 12 - New soil chemical testing technologies and recommendations for agricultural and environmental benefit.

Paul Kennelly will lead session 20 - Laboratory QA and assessment: A key determinant of a successful soil science consultancy

On Wednesday 21st November, ASPAC will be running a workshop (see below), and this will be followed by the ASPAC AGM from 4pm.

Workshop Title - Soil and Plant Analysis Workshop – new thinking, extra value

Date: Wednesday 21 November 2018, **Time**: 1330-1530

Venue: Hyatt Hotel Canberra

Cost: Complimentary.

The workshop structure will include both presentations and group interaction. There will be three Managers from major soil and plant testing facilities that cover commercial, government and university sectors. Each will give their perspective of existing and emerging techniques and tests, future challenges and the value their laboratory offers to the soil and plant testing Industry. New tests will include recent advances in environmental testing such as bio-available nutrients in sediments moved off site along with tests for 'fingerprinting' sediment source. A fourth presenter will discuss sampling and interpretation of results for soil fertility purposes. The final presenter will demonstrate improvements in the quality of both soil and plant testing due to training programs offered by ASPAC

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for example, and due to third party assessment of laboratory competency by NATA (National Association of Testing Authorities, Australia) and by ASPAC.

ASPAC is sponsoring a Soil Judging Competition on the weekend preceding this conference.

Travel Awards 2018

2018 Janice Trafford ASPAC Travel Award

This year's Travel Award of $1,500 has been granted to Oxana Belyaeva. Oxana is currently employed as a soil research scientist in the School of Veterinary and Agricultural Science, the University of Melbourne. She holds a Masters degree in Agrochemistry and Soil Science and a PhD in Soil Science, which were awarded in 1996 and 2002 respectively, from the Rostov State University, Russia. Her area of research specialty is general soil fertility with a slant towards plant and soil nutrition in different agricultural systems. Oxana's research projects included implementing and developing applied technologies for resolving of the range of problems devoted to improving the quality of crops (mainly grain crops) and soils.

Oxana will present an oral paper "Impact of urease and nitrification inhibitors on ryegrass productivity in the high rainfall zone of southern Australia" at the 21st World Congress of Soil Science (21st WCSS).

ASPAC certification of laboratories for soil and plant elemental tests - how it is assessed and what does it mean.

The Australasian Soil and Plant Analysis Council (ASPAC) runs inter-laboratory proficiency programs (ILPPs) to assess laboratory proficiency for a wide range of soil and plant chemical tests. ASPAC Certification (not accreditation) is granted for an individual test when a laboratory demonstrates satisfactory ILPP performance for that test.

Members of ASPAC received newsletter articles recently, that showed clear evidence of improvements in the performance of soil and plant testing since inception of the ILPP's in 1995, but more so in the last 10 years. This can be attributed to both the proficiency and training programs run by ASPAC, as well as the desire for formal recognition of technical competence through involvement in the certification and accreditation processes on offer.

ASPAC's criteria for certification

In an ILPP, a selection of carefully prepared samples is sent to participating laboratories to compare their method-by-method measurement performance relative to those of their peers across Australasia. The process is method-specific, as each test is assessed separately using internationally-respected non-parametric statistics. Obviously, the peer review process is strongest for methods with most participants, always ≥7 and typically well in excess of that number. Regular feedback with "round-by round" regularity provides tangible evidence to guide laboratory managers in their efforts towards measurement excellence.

Eligibility for ASPAC certification is achieved by demonstrating (over 3 proficiency rounds) the ability to consistently present results within an accepted range of the assigned value (usually the median of participants' results). The range of accepted values is determined by the ASPAC score (analogous to the z-score). If the score for any result is >2 after statistical calculations have been applied to the raw data, that result is then excluded from the data set; it is deemed an outlier and the laboratory incurs two demerit points for that test. A second iteration is run and new

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statistical parameters (median, median absolute deviation, score) are calculated from the remaining data. Any new result where the score >2 is deemed a straggler and the laboratory incurs one demerit point for that test.

To be eligible for certification in any method, a laboratory must have no more than four demerit points incurred across the three proficiency rounds (totalling 12 samples) in the annual program. If a result is not submitted for any sample, two demerit points are automatically incurred. The maximum number of demerit points a laboratory can accrue from the four samples in any single round is three (of a possible 8). So if a round is missed or systematic error results in more than three points in any round, ASPAC have decided that the laboratory will still have the opportunity to achieve certification, if they perform well in the other two rounds. Following the conclusion of the annual series of samples, laboratories with ASPAC membership will receive a signed PDF certificate stating the methods for which the laboratory has received ASPAC certification. Approximately two weeks after the certificates are issued to laboratories, ASPAC updates its website (see www.aspa.-australasia.com) with the list of certified elements and methods for each of its member laboratories.

What ASPAC certification means

To understand what certification means, one should take into account how the ILPPs are run and what ASPAC asks participants to do. ASPAC asks participants to:

- Analyse soil samples as received - they have been air dried, ground to <0.5 mm, mixed and tested to show the samples are homogenous.
- Re-dry plant samples @ 65°C to constant weight. These too have been fine ground after drying and tested to show they are homogenous.
- Test the proficiency samples exactly as you would for your routine day to day chemical analysis. Only conduct tests that are routinely performed and only test in duplicate if that is the normal procedure.

Global Proficiency (ASPAC's proficiency provider) outsource the preparation of ILPP plant and soil samples to an ASPAC approved laboratory, that does drying, grinding, mixing and testing for homogeneity. Samples are then dispatched to participating laboratories. Sample preparation has already been done for participants – there is no need to do anymore to the samples except for plants, which need to be re-dried at 65°C to constant weight. To ensure homogeneity (most important for proficiency testing) soils are ground to a finer specification (<0.5mm) than laboratories should use for most routine soil tests (<2 mm). Plants similarly are ground to a fine particle size to ensure homogeneity.

For routine soil testing, <2 mm infers the entire soil sub-sample has been ground or rolled to pass a 2mm sieve. Soil test calibrations (and interpretation standards for nutrients in soil) have mostly been based on this particle size. By asking participants to analyse samples as received, within laboratory variations (error) in sample preparation are not covered/assessed in the certification process. Instead emphasis is on within laboratory performance in undertaking the extraction/digestion procedure and the analytical finish used for each test. Significant errors can occur during routine soil and plant sample preparation. These include contamination, over-grinding and incomplete grinding (of the entire sub-sample) and mixing that result in the final sample not being representative of the original sample taken in the field. Grinding soil to a much finer particle size (e.g.<0.1mm) increases the surface area of the soil that will be in contact with the extractant or digest solution, resulting generally in higher elemental concentrations. For plants, particle size is less critical as most plant tests are total elemental determinations.

Certification of proficiency for a test is achieved when a laboratory demonstrates satisfactory performance - not exceptional, not very good, but satisfactory performance. Laboratories should do exactly as ASPAC requests i.e. – treat the ILPP samples the same as is done for their routine samples. By doing so, a laboratory will get full value

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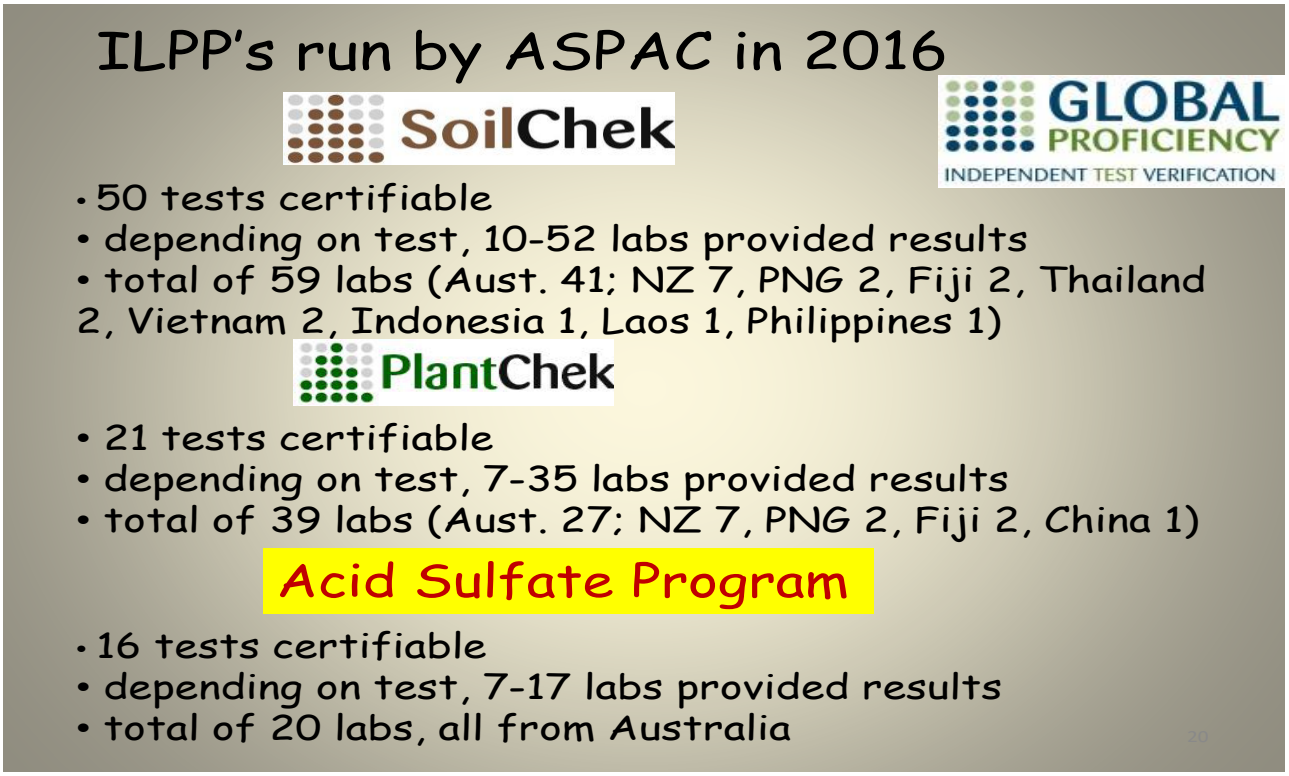
out of the ILPP's by testing out the robustness of its routine procedures and batch quality control safeguards. If good performance is achieved over time, a laboratory will have more confidence in the accuracy of its routine analytical systems. A laboratory can record eight demerit points from four samples in one round, and a straggler from one of the other eight samples (from the two other rounds) and still gain certification of the test for that calendar year. So there is plenty of scope to do exactly as ASPAC requests. Quality improvement comes from making mistakes, trouble shooting, taking corrective action then documenting what happened. All laboratories make mistakes - it is what they do to minimize recurrence that leads to excellence in routine measurement performance.

The following diagram shows the ILPPs overseen by ASPAC in 2016. It is so pleasing to see the number of participating laboratories increase over the years, as have the number of tests in each program. Special thanks to Global Proficiency for their assistance here. Global Proficiency, who are based in Hamilton New Zealand, have been ASPAC's contracted proficiency provider since 2004.

Difference between Certification and Accreditation of tests

It is the responsibility of laboratory management to pay close attention to total quality management. This involves taking account of factors such as: proficiency performance (which ASPAC certification is based on) technical competence and procedures, sample preparation, records of corrective actions, customer complaints, instrumental accuracy checks and maintenance, staff training/qualifications, standard-solution preparations, method validation/verification, internal audits, batch quality control, reports to clients, etc. Laboratory accreditation to ISO-IEC 17025 standard covers all of these. The National Association of Testing Authorities (NATA) is responsible for laboratory accreditation and compliance in Australia. In effect, certification is a subset of accreditation, considering only one (arguably the most important) of the factors assessed for accreditation.



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Exchangeable Cations Method Selection

Overview

The major alkali exchangeable soil cations, Na^+, K^+, Mg^{2+} and Ca^{2+}, profoundly influence soil structure and fertility, so measuring and managing their concentrations is an important part of our stewardship of the soil resource.

Exchangeable Soil Cations and Cation Exchange Capacity

Soil cations are termed exchangeable if they can be displaced from exchange sites (e.g. negatively charged sites on clays, oxide minerals and organic matter) on the soil by a solution containing a large excess of another cation, e.g. NH_4^+.

The concentration of exchangeable cations able to be held by a soil is limited by the number of exchange sites, which in turn is controlled by clay mineralogy and other soil structural and chemical factors. This intrinsic soil property of a soil is termed its cation exchange capacity (CEC).

Traditionally, CEC is quantified by measuring how much of the displacing cation (e.g. NH_4^+) the soil retains at the fixed, high pH (7–8) of the cation extracting solution. This approach works for soils with 'fixed' or 'permanent' charge, i.e. the charge inherent in the clay minerals by virtue of impurities or defects in the aluminosilicate day lattice.

However, many soils are acidic, and their exchange capacity is due at least in part to colloids that have functional groups on their surfaces whose charge varies with pH, e.g. organic matter and sesquioxides. In these soils, acidification progressively replaces the exchangeable Na^+, K^+, Mg^{2+} and $Ca^{2+}$by acidic cations such as Al^{3+}, and in some soils by Mn^{2+}, Fe^{2+} and H^+ (Slattery et al. 1999).

For these acidic soils, the CEC measured at a high pH using the traditional approach will not reflect the true (or effective) cation capacity of the soil at its natural pH in the field. Consequently, a second term, effective CEC (ECEC), is used to describe the cation exchange properties of acidic soils. In method 15J1, Rayment & Lyons (2011) calculate ECEC as the sum of the displaced Na^+, K^+, Mg^{2+} and Ca^{2+}, plus exchange acidity, where exchange acidity is measured in a 1 M KCl solution (method 15G1). However, other methods of measuring exchange acidity, and of calculating ECEC, may be in use by different laboratories.

Measuring Exchangeable Soil Cations and Cation Exchange Capacity

In the laboratory, either the amount of the displacing cation that is retained by the soil (CEC approach), or the displaced cations themselves (ECEC approach), can be measured. Test methods to measure ECEC and CEC are simple for soils that do not contain additional sources of the cations Na, K, Ca and Mg. However, the presence of soluble salts (e.g. NaCl, gypsum and calcium carbonate) can complicate the measurement of exchangeable cations and CEC/ECEC in some soils, since these substances have the potential to inflate cation and CEC results. More complex test methods are used to minimize the influence of these additional cation sources on ECEC and CEC measurements. The principles of the methods more commonly used in Australia, and their pros and cons, are described briefly below and in detail in Rayment & Lyons (2011).

The customary solution used in Australia to displace cations is 1 M ammonium chloride (NH4Cl), which extracts both soluble and exchangeable cations. The presence of salinity and gypsum in soils can be detected as they result in an elevated electrical conductivity (EC) of the 1: 5 (w/v) water extract. For soils with an EC1:5 >0.3 dS/m, Rayment & Lyons (2011) recommended that the soluble salts are washed out before the exchangeable cations are displaced.

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The presence of gypsum in an extract with a high EC can be detected as (white) $BaSO_4$ using a spot test. It can be further confirmed by elevated sulfate results in a 0.01 M $Ca(H_2PO_4)_2$ extract. The approach for (alkaline) gypsiferous soils is to combine the pre-wash (to remove soluble salts) and to substitute much of the water in the displacing solution with alcohol, to suppress the solubility of any remaining gypsum.

Lastly, lime ($CaCO_3$) and/or dolomite may be present if the pH in the 1:5 (w/v) calcium chloride solution is >7.0. Again, the solubility of these salts is minimised by using an alcoholic medium, and by raising the solution pH to 8.5. (The displacement method used affects the cation results, so it should be reported along with the results).

The appropriate cation method for the soil and management goals can be selected according to SSA 2013, or by working through **Fig. 1** following.

Limitations

Cation-related measurements are fundamental for soil classification and the definition of clay type. Monitoring of exchangeable cations provides valuable insights into system function, e.g. increasing exchangeable Na percentage (ESP) or exchange acidity in the profile may indicate system imbalances, and exchangeable K is a useful indicator of K supply for crops. However, monitoring trends in both soil and plant composition provides superior insights into how a system is functioning.

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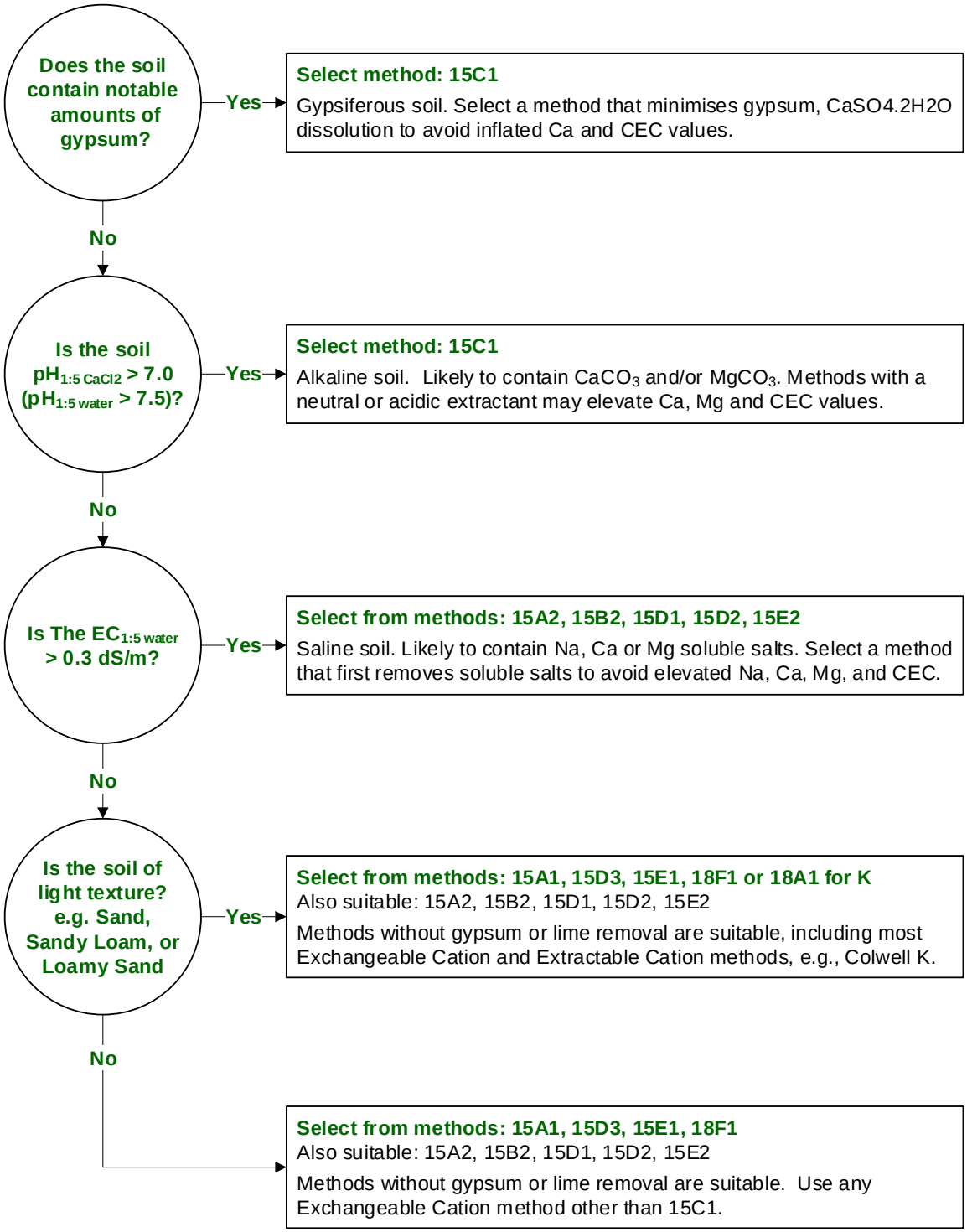

Figure 1. Exchangeable Cation method selection, using method codes from Rayment & Lyons (2011). Adapted from: Soil Science Australia (2013). Measuring Soil Cation Exchange Capacity and Exchangeable Cations. Soil Science Australia. The method codes are described briefly in Table 1.

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Code	Method Description
15A1	1M NH_4Cl at pH 7.0. No pre-treatment for soluble salts.
15A2	1M NH_4Cl at pH 7.0. Pre-treatment for soluble salts.
15B2	1M NH_4Cl at pH 7.0. Filtering/washing for cations and leaching for CEC. Pre-treatment for soluble salts.
15C1	Alcoholic 1M NH_4Cl at pH 8.5. Leaching to remove soluble salts and subsequently for exchangeable cations and CEC.
15D1	1M NH_4OAc at pH 7.0. Leaching to remove soluble salts and subsequently for exchangeable cations and CEC.
15D2	1M NH_4OAc at pH 7.0. Automated extractor.
15D3	1M NH_4OAc at pH 7.0. No pre-treatment for soluble salts.
15E1	0.1M $BaCl_2$/0.1M NH_4Cl. No pre-treatment for soluble salts.
15E2	0.1M $BaCl_2$/0.1M NH_4Cl. Pre-treatment for soluble salts.
15G1	1M KCl exchangeable acidity ($H^+ + Al^{3+}$).
15J1	Effective cation exchange capacity (ECEC). Summation of Na^+, K^+, Mg^{2+}, Ca^{2+} and exchange acidity (15G1).
18A1	0.5M $NaHCO_3$ at pH 8.5. Extractable K (Colwell).
18F1	Mehlich 3 Extractable elements: P, Ca, Mg, Na, K, Fe, Cu, Mn, Zn, B, S, Al.

Table 1. Exchangeable Cation method codes from Rayment & Lyons (2011).

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